

Chemistry Safety Notes

Volume 3, Issue 6

September 2015

"Chemistry Safety Notes" is published by the Chemistry Dept. Safety Committee, written & edited by Debbie Decker, Safety Mgr.

ACS Program In A Box "Tales of Lab Safety: How to Avoid Rookie Accidents" October 20, 4:00PM Rm. 172 All are invited!

There is a learning curve with everything, but mistakes made in the lab can ruin your research and cause dangerous accidents. A lab fire at the University of California, Los Angeles, in 2008 led to the death of researcher Sheharbano (Sheri) Sangji, shocking the chemistry and laboratory safety communities. Details of the incident and its cultural and legal aftermath will be discussed, along with other recent notable chemistry lab incidents and their common themes.

What You Will Learn:

- What lab incidents have happened recently
- What common factors underlie those incidents
- What are some of the resources available to help people work safely

The Chemistry Club has graciously facilitated Department registration for this event. The presenters, Jyllian Kemsley, Senior West Coast Editor of C&EN, and Mary Beth Mulcahy, Chemical Safety Board Investigator, are colleagues and friends of mine and the webinar will be very good.

Legacy Waste

Last month, we had another potential issue with legacy hazardous materials—several reagents, originally stored under diethyl ether, were discovered in a back cabinet, all dried out and crusty. Segregation in a fume hood and a quick call to EH&S solved that issue.

PLEASE continue to be diligent about reviewing inventory and checking those out-of-the-way spots in the lab for forgotten bits.

Remember—hazardous waste disposal remains free of charge until June 30, 2016.

Risk Assessment

Earlier this month, most of the lab safety representatives attended an advanced workshop on hazard and risk assessment. The facilitator provided a number of options, tools, and methodologies to accomplish assessments. Very interesting.

I'm discussing with the Chair about how to encourage the Department to move from a compliance culture to a culture of risk management. Stay tuned to see how we can accomplish a shift like this. If you have ideas or would like to use some of the materials provided, drop on by.



Debbie's Moving -

From Chemistry 122 to Chemistry Annex 3467, effective on or about October 20th. Come by for a visit!



Building Project Update

The general updates are as follows:

- **Chemistry Rooms 1, 3, & 92 Remodel (chilled water loop)** – Project is launching next week. Check the bulletin board across from 122 for project drawings and schedule.
- **Chemistry Safety Improvements** - We are finalizing the bid alternates and will get that out to bid by the end of this month with construction scheduled to start in December.
- **Chemistry Seismic and Life Safety Corrections** – We are kicking off the design development phase with the consultant team with that phase anticipated to be completed in November.
- **Chemistry Discovery Complex** – The Project Vision Committee (Academic vision) is scheduled for a kick-off meeting at the end of September with recommendations due to the provost by the end of December.

Gabby Nepomuceno shares the following post from the Chemjobber blog:

The 200th Hour*

Derek Lowe has his *Chemistry World* column up on graduate school and academic chemical safety and [he makes a great point about experience levels](#):

...That vivid memory illustrates another aspect of the problem: just as lab hours per calendar day (and perhaps also hours worked alone) are at their highest, one's own experience is nowhere near at its peak. Graduate school is where chemists encounter a lot of reagents and procedures for the first time, and not all of these encounters will go smoothly. This is when one might find out, for example, just how remarkably air-sensitive trimethylaluminum is (if there isn't a flame burning from the end of the syringe needle, the bottle has probably gone off), or just how long a large aqueous phosphorus oxychloride workup can sit around before it suddenly erupts all over the inside of a fume hood (several hours, damn it all)....

I'm reminded [an episode of one of my favorite old shows "The Unit,"](#) where the youngest operator on the team grazes a senior operator in the arm during a training exercise. After giving Bob a thorough razzing, they congratulate him on reaching his "200th hour" where he has "enough experience to be confident, enough to screw up real good."

It is a funny aspect of graduate school in chemistry where you're the least experienced, you work the most hours that you'll probably work during one's time in research chemistry and you're likely to be the least supervised you'll ever be (if you continue onto industrial bench chemistry).

*Apparently this is flying terminology?

Editor note: I think this is important to keep in mind as we welcome our new graduate student researchers and undergraduates into laboratory. Please avoid working alone and at odd hours where there are few people around to come to your aid if something bad should happen.

A little birdie sent the following tidbit to me. Here's the link to the original reddit article:

https://www.reddit.com/r/tifu/comments/3lb11d/tifu_by_making_and_detonating_a_bomb_at_school/

Here's the (less-interesting but still accurate) news account:

<http://publicsafety.tufts.edu/ehs/files/March2015.VII-LabAccidentsExplosionsInvolvingNitricAcid.pdf>

Making and Detonating a Bomb at School

Like almost all TIFUs this did not happen today, but some time ago. For obvious reasons I am using a throw away and being vague about some details because anyone who reads this and is a chemist at the university I attended will know who I am. For some background, I was a graduate student at a large R1 university getting a Ph.D. in chemistry. As an organic chemist, I frequently use compounds that require delicate handling and an exquisite extravagance of attention. I was working in a lab that frequently used fairly reactive things (in that they reacted spontaneously and spectacularly with air or water)¹. The problem with that is when you use dangerous things every day, you get used to them, and the danger goes away. You become complacent. Maybe you make assumptions about the what safety protocols others are following. This is a horrible practice.

One of the things I frequently used to clean certain types of glassware was concentrated nitric acid. You really need to be careful with nitric acid, not only is it a very strong acid, but also insanely good at nitrating things in addition to being a superb oxidizing agent. This means that not only is it super corrosive and can eat through copper and other metals pretty quickly, but that it also reacts violently and often explosively with most organic chemicals to produce toxic gas + a nitrated compound.² Nitrated compounds make great explosives. As Wikipedia says, "Nitration of organic compounds with nitric acid is the primary method of synthesis of many common explosives, such as nitroglycerin and trinitrotoluene (TNT). As very many less stable byproducts are possible, these reactions must be carefully thermally controlled, and the byproducts removed to isolate the desired product."³

I'm sure you can see where this is going...

So here is where the chain of events that was my f*up begins. I was cleaning some glassware with nitric acid, which is a fairly common method to get very clean glass.^{4,5} The waste bottle that we use to dispose of nitric acid was full, so I had to procure a new empty bottle to use as the nitric acid waste. Typically we use an empty bottle of nitric acid as the waste container for used nitric acid. This way, the nitric acid is going into a container that only ever held nitric acid (IE no random organic chemicals left around in the bottle to react with the acid). There were no empty nitric bottle in lab so rather than go get a new one 4 floors down, I grabbed a common use waste bottle. These are 4 liter glass bottles with a screw on cap.⁶ Usually they are used to collect organic waste, brought to a central facility where they are emptied and then thoroughly cleaned. University protocol is that they are first cleaned with ethanol, then water. The idea is that the only remnants in these bottles should be water. I happened to pick a bottle that had not been washed with water. Knowing that nitric acid was dangerous, I visually checked the bottle to make sure it was empty. There was a little bit of water (or so I thought)⁷ in the bottom, which did not concern me because nitric acid and water are fine to mix.⁸ I proceeded to clean my glass using a total of 30-50 mL of nitric acid, which I disposed in the waste container. Knowing that nitric acid could react with organics, I left the waste bottle un-capped in my fume hood for about 60 seconds after I put the nitric in. Seeing no reaction, I then capped the waste bottle loosely. This probably saved me a trip to the hospital.

Now, the astute chemist reading this may have figured out what happened next.⁹ Nitric acid and ethanol (remember this bottle was supposed to be washed with water, but never was) react very violently to produce heat and a large amount of gas. This reaction has an incubation time of a few minutes before it really kicks in. So 20 or so seconds after capping this bottle, I hear an ominous whistling sound. The kind of whistling you would rather not hear in a chemistry lab. I look at my fume hood and saw a very large and copious amount of brown gas (NO_x) billowing out from my loosely fitted cap. As the whistling increased to a truly terrifying pitch, I had a few seconds to dive behind a wall before the waste bottle exploded with a force much larger than that mortar from the front page yesterday.^{10,11,12}

Here I f*ed up again as despite my 10 or so second lead time, I did not warn anyone that a glass shrapnel bomb was about to go off. I am so f*ing lucky that no one decided to come around the corner at that moment. As the nitric acid tinged glass rained down upon me, my lab mates rushed to see what was wrong. I yelled for them to evacuate the lab as a billowing cloud of brownish green gas (a toxic mix of nitric acid, nitrous oxide, ethyl nitrate and the various other chemicals in my hood which were vaporized and atomized) was spewing forth from my fume hood. Alarms were going off, lights in the ceiling were blown out and haphazardly hanging from their sockets, I'm pretty sure an undergraduate was crying...

Needless to say, we exited the lab in admirable time. A few minutes later, the chief safety officer arrived with gasmasks in tow. Our lab replaces the air about every 2 minutes due to the fume hoods and by design for instances just like this, so after 5 minutes, we deemed it safe enough to reenter with gasmasks on. The level of destruction was actually surprising.¹¹ Everything in my hood was destroyed. The window directly behind my fume hood was destroyed. That window was made out of ¼ inch thick safety glass. This explosion sent a shard or shards of glass flying hard enough to bust a hole clean through 1/4 inch thick safety glass... Had I been standing in front of this thing, or had anyone else, they would have been in the hospital with some very serious injuries. In doing some research, I found out this is not a fairly uncommon laboratory accident and a simple google search of Nitric acid + ethanol furnishes a number of safety reports on similar incidents.

An investigation found out that two parties were at fault. The waste bottle should have never had ethanol left over in it. Improper handling on EH&S was determined to be the major cause. Me being a f*rd was determined to also be a cause. Because of this, a number of safety protocol with how waste bottles are handled were changed, and incoming graduate students get to hear about what I did. Gratifyingly I did not get in trouble because everyone handled themselves like adults.

TLDR: I might be the only person to use the excuse "I blew up my lab today" for why I was late to my first date with my future fiancé and have it be a real excuse. *Luckily she realized I was a keeper, is now my fiance and we have a great first date story.

The damage <http://imgur.com/a/AoRpm>

References:

1. <https://www.youtube.com/watch?v=EmkBH-ncG1Y>
2. Nitric acid reaction with protective gloves. <https://www.youtube.com/watch?v=aBVdGGml6bU>
3. https://en.wikipedia.org/wiki/Nitric_acid
4. <http://curlyarrow.blogspot.com/2008/01/how-to-clean-your-sintered-funnel.html>
5. http://www.starna.com/ukhome/d_tech/tech01.html
6. <https://static.fishersci.com/images/F203653~wl.jpg>
7. It should be noted that working in an organic lab all day removes your ability to smell ethanol and most other solvents.
8. Have PhD, trust me.
9. <https://www.youtube.com/watch?v=uFwiZYfEsuY>
10. <http://i.imgur.com/sHhftlF.gifv>
11. <http://publicsafety.tufts.edu/ehs/files/March2015.VII-LabAccidentsExplosionsInvolvingNitricAcid.pdf>.
12. A similar explosion albeit in a smaller non glass bottle, with less toxic things ingredients. <https://www.youtube.com/watch?v=ulNeRQxORTM>

Edit: not safety glass, should have fact checked that and referenced it.

Also, this did not happen at your university. Waste bottle over pressurization is not an uncommon lab accident.